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XXXIX. *Chemical Experiments and Observations on Lead Ore.* By Richard Watfon, D. D. F. R. S. in a Letter to Sir John Pringle, Bart. P. R. S.

TO SIR JOHN PRINGLE, BART. P. R. S.

S I R,

Cambridge,  
June 13, 1777.

Read July 9,  
1778.

THE following experiments and observations, it is apprehended, will not be thought uninteresting by persons versed in chemistry. May I beg the favour of you to communicate them to the Royal Society?

I am, &c.

LEAD ORE, as dug out of the mine, is generally much mixed with spar, lime-stone, and other substances, bulk for bulk, heavier than the ore itself. It undergoes various dressings before it becomes a merchantable commodity, the general tendency of which is to free it, as much as possible, from every heterogeneous impurity.

Suppose

Suppose that a cubic foot of lead ore, which contained no spar or other extraneous matter, would weigh 7800 ounces, and that a cubic foot of spar, which contained no lead ore or other foreign substance, would weigh 2700 ounces, then would a mixture, consisting of a cubic foot of pure lead ore and a cubic foot of pure spar, weigh 10500 ounces, and one cubic foot of such a mixture would weigh 5250 ounces. It is obvious that, according to the different proportions in which the particular kinds of spar and lead ore here assumed are supposed to be mixed together, a cubic foot of the mixture will have different weights, the limits of which are on the one hand 7800, and on the other 2700 ounces; it never can weigh so little as 2700 ounces, for then it would consist intirely of spar without any lead ore; nor can it ever weigh so much as 7800 ounces, for then it would consist intirely of lead ore without any spar.

From this view of the matter it is evident, that the purchasing of lead ore by the measure, which is the general though not the universal custom in Derbyshire, is a mode liable to some exception; since a dish, containing any definite measure, must have different weights, according as the ore with which it is filled is more or less free from spar. And it is scarce possible, by repeated dressings,

dressings, to separate all the spar from an ore, or equal portions of it from equal portions of ore.

There is a diversity, however, in the weights of equal measures of lead ore, which probably does not arise from sparry or other heterogeneous accretions, but from the nature of the ore itself. I have carefully taken the specific gravities of many of the Derbyshire lead ores; the weight of a cubic foot of the lightest which I met with was 7051 ounces, and the weight of a cubic foot of the heaviest was 7786 ounces; the difference amounting to between a ninth and a tenth part of the weight of the lightest. There are probably other ores of lead which differ more in their specific gravities than these here mentioned; but the difference between these is sufficient to shew the great uncertainty of purchasing lead ore by the measure, since ten dishes of one sort of ore may not weigh more than nine dishes of another sort, though both the sorts be equally well dressed.

Lead ore is not always of the same goodness in the same mine, nor even in the same part of the same mine; and, what is more remarkable, the different parts of the same lump of ore have different specific gravities. I could not easily have believed this, unless a variety of experiments had convinced me of the fact.

They were employed this year at Holywell in smelting a lead ore from the Isle of Man; the ore was rich in silver. A lump of this ore, weighing about ten ounces, was broken into several pieces, and such of the pieces were selected as appeared to the eye to be wholly pure. By taking the specific gravities of six of these pieces I found, that a cubic foot of the lightest kind would have weighed 6565 ounces, and a cubic foot of the heaviest kind would have weighed 7636 ounces. Supposing the specific gravity of water to be denoted by 1,000, the mean specific gravity of the six different pieces of this ore may be expressed by 7.115.

A very pure specimen of tessellated lead ore, from a mine near Ashover in Derbyshire, was broken into six pieces, weighing near one ounce each. A cubic foot of the lightest of these pieces would have weighed 7326 ounces, and a cubic foot of the heaviest would have weighed 7786 ounces. The mean specific gravity of the six pieces was 7.566.

At the same mine they frequently meet with small quantities of steel-grained lead ore. Six different pieces of the same lump of this kind of ore were chosen, each of which appeared quite free from spar and every other impurity. A cubic foot of the lightest of these pieces would have weighed 7188 ounces, and a cubic foot of

the heaviest would have weighed 7442 ounces. The mean specific gravity of the six pieces was 7.342.

Other lumps of ore, from different mines, were respectively broken into different pieces, and scarcely any two pieces of the same lump were observed to agree in their specific gravities. This diversity in the specific gravities of the several pieces of the same lump of ore may be owing, either to the different proportions in which the constituent parts of the ore are combined in the several pieces; or to the different quantities of extraneous substances imperceptibly mixed with them, or, which seems most probable, to a diversity in the size or configuration of these pores.

But be the cause of the diversity in the specific gravities of different pieces of the same lump of ore what it may, the fact, I believe, is certain, and by no means singular; for not to mention the varieties observable in the specific gravities of different pieces of roll brimstone, of corrosive sublimate, of cast steel, and other factitious substances, the natural spars generally found along with lead ore are subject to a similar diversity, though not perhaps in an equal degree.

A piece of rhomboidal, otherways called refracting or lantern spar, was broken into four smaller pieces, the specific gravities of which were 2.675, 2.687, 2.715,

2.723; the medium of the four is 2.700. Mr. COTES fixes the specific gravity of Iceland crystal at 2.720, and WALLERIUS fixes it at 2.700.

The specific gravities of four pieces of the same lump of cubical spar were 3.204, 3.218, 3.222, 3.231; the medium of the four is 3.219.

Six ounces of fine tessellated lead ore were put into a crucible and exposed, at first, to a gentle, afterwards to a strong fire: the ore grew red, and emitted fumes which smelled of sulphur; at length it melted, and the fumes became very copious; they were accompanied with a yellowish flame upon the surface of the melted ore, and when collected had a whitish appearance. The crucible after the ore had continued a full hour in perfect fusion was taken from the fire, and when it was cold it was broken. The mass which it contained weighed five ounces and an half; there were no scoria observable on its surface, nor were any particles of metal formed, it was still an ore of lead.

The mass remaining from the last experiment was put into a fresh crucible, and exposed to a strong melting heat; the fumes which arose from it seemed to be heavy; they brooded over the surface of the melted mass in undulating flames, which now and then appeared like burning zinc. The lead was now formed, and many  
particles

particles of it were sublimed to at least six inches above the surface of the liquid in the crucible. After letting the crucible continue two hours in this state, I poured out its contents, and found them consisting partly of lead, partly of lead ore, and partly of a very minute portion of brownish scoria.

These experiments prove, that some substance or other is contained in lead ore, which must be dispersed before the ore can be formed into lead; and they shew too, that it requires a considerable time to effect the dispersion of this substance, since six ounces of ore, though kept three hours or more in complete fusion, were not wholly brought into the form of lead; and, lastly, they render it probable, that the fumes, arising from melted ore, carry off with them no inconsiderable portion of the lead itself. At the great smelting houses in Derbyshire, they put a ton of ore at a time into the furnace, and work it off in eight hours; the ore might be wholly melted in one hour, but the lead perhaps is not formed in the greatest possible quantity in eight hours.

Some fine tellurated lead ore from Derbyshire was pounded into small lumps, each about the size of a pea, and carefully picked from spar and other impurities. Sixteen ounces of this ore, thus previously cleaned, were distilled in an earthen retort; as soon as the ore felt the



fire, the stopple of the quilled receiver had a strong smell resembling that of the inflammable air separable from some metals by solution in acids; soon after a small portion of a liquid came over into the receiver; the fire was then raised till the retort was of a white heat, when a black matter began to be sublimed into the neck of the retort; the operation was then discontinued. This experiment was undertaken with a view of seeing whether sulphur could be separated from lead ore, as it may be from some species of the pyrites, by distillation, and it appears from the issue of the experiment that it cannot. What might have been the event of the experiment if it had been conducted with a very gentle heat for a long time, I cannot pretend to say. Upon breaking the retort I found, that the ore had been melted during the operation, for there was a consistent cake of ore of the figure of the bottom of the retort; the weight of this cake was fifteen ounces and an half, the weight of the liquid in the receiver, and of the black matter which had been sublimed, did not together amount to one quarter of an ounce, so that a quarter of an ounce or more had been dispersed, probably in the form of air, or some elastic fluid. The ore by this process had lost one thirty-second part of its weight; but I am of opinion, that if the operation had been conducted with a less degree of heat and  
continued

continued for a longer time, the quantity of liquid would have been augmented. The liquid did not effervesce with either acids or alkalies; nor did it produce any change in the colour of blue paper, though I am certain, from experiment, that one drop of oil of vitriol, though diluted with two ounces of water, would have produced a sensible redness on the blue paper which I used. The liquid, notwithstanding, had an acid taste, and a pungent smell, resembling that of the volatile vitriolic acid. The experiment ought to be repeated with a larger quantity of ore, in order to ascertain the quantity and quality of the liquid, separable therefrom by simple distillation. The black matter which had been sublimed into the neck of the retort, was examined with a microscope, and it appeared to be pure lead ore; hence it is probable, that by a due degree of heat in close vessels, lead ore might be entirely sublimed without being decomposed; for the melted ore which was found at the bottom of the retort, had not any appearance of either scoria, or of lead, upon its surface. Finding that sulphur could not be separated from lead ore by distilling it without addition, and yet being much disposed to think, that it contained a considerable portion of sulphur, I first thought of distilling it with charcoal dust, iron filings, sand, and other additions; but recollecting that sulphur might be separated from anti-

mony

mony by solutions in acids, I thought it not improbable, that it might be separated from lead ore by the same means, and the success of the following experiment abundantly justified the conjecture.

Upon ten ounces of lead ore, cleansed as in the preceding experiment, I poured five ounces of the strongest fuming spirit of nitre; this strong acid not seeming to act upon the ore, I diluted it with five ounces of water; a violent ebullition, accompanied with red fumes, immediately took place; the solution of the ore in this menstruum became manifest, and when it was finished, there remained floating upon the surface of the menstruum a cake of fine yellow sulphur, perfectly resembling common sulphur.

I repeated this experiment a great many times, in order to ascertain the quantity of sulphur contained in lead ore, and separable therefrom by solution in acid of nitre. The results of different experiments were seldom the same: the matter separable from the ore by solution, after being repeatedly washed in large quantities of hot water, in order to free it from every saline admixture, sometimes amounted to more, sometimes to less than one-third the weight of the ore. This matter may, for the sake of distinction, be called crude sulphur. Its apparent purity might induce a belief that it contained no  
hetero-

heterogeneous mixture, yet the following experiments shew how much we should be deceived in forming such a conjecture, and how rightly it is denominated crude sulphur.

From one hundred and twenty parts, by weight, of lead ore, I obtained, by solution in acid of nitre, subsequent washing in hot water, and drying by a gentle fire, forty parts of a substance which looked like sulphur: these forty parts were put on a red-hot iron, the sulphur was made manifest by a blue flame and pungent smell. When the flame went out, there remained upon the iron unconsumed twenty-six parts of a greyish calx; the weight of the sulphur which was consumed must therefore have amounted to fourteen parts, or between one eighth and one ninth part of the weight of the ore. It has been observed, that the weight of the matter, separable from lead ore by solution in acid of nitre, sometimes exceeded, and sometimes fell short of, one third part of the weight of the ore; this variety, as far as I have been able to observe, does not extend to the quantity of sulphur contained in a given quantity of ore, but depends upon the quantity of calx remaining after the burning of the sulphur. Different lead ores will, doubtless, contain different quantities of sulphur; but that the sulphur contained in the lead ore which I examined constitutes between one eighth and one ninth part of the weight of

the ore, is a conclusion upon which, from a variety of experiments, I am disposed to rely.

There are annually smelted in Derbyshire about ten thousand tons of lead ore: now if means could be invented (which I think very possible) of saving the sulphur contained in ten thousand tons of ore, supposing that the ore should only yield one tenth of its weight of sulphur, though it unquestionably contains more, Derbyshire alone would furnish annually one thousand tons of sulphur, the value of which would annually be about fifteen thousand pounds. I mention this circumstance thus publicly, in hopes that the lead smelters may be induced to prosecute the object. If the sulphur contained in lead ore could be collected, it would not only be a lucrative business to the smelters, but a great saving to the nation. We at present import the sulphur we use, and the consumption of this commodity is exceeding great, in the making of gunpowder, in forming the mixture for covering the bottom and sides of ships, and in a great variety of arts. The smelters need not be apprehensive lest the quality of the ore should be injured by extracting the sulphur. Eighteen hundred weight of ore, from which the sulphur has been extracted, will certainly yield as much lead as twenty hundred weight of ore, from which the sulphur has not been extracted, and it will probably yield more.

Arsenic

Arsenic is extracted from a particular ore in Saxony, by roasting the ore in a furnace, which has a long horizontal chimney; the chimney is large, has many windings and angles, that the arsenical vapour which arises from the ore may be the more easily condensed: the arsenic attaches itself like soot to the sides of the chimney, and is from time to time swept out. It is very probable, that by some such contrivance the sulphur contained in lead ore might be collected. The smelters call everything sulphur which is volatilized during the roasting or fluxing of an ore; but none of those with whom I have conversed had any notion that common sulphur could be separated from lead ore.

The greyish calx which remained upon the iron after the sulphur was consumed, was put upon a piece of lighted charcoal; the heat of the charcoal being quickened by blowing upon it, a great number of globules of lead were formed upon its surface. From hence it appears, that this calx is not an unmetallic earth contained in the ore, which the acid of nitre could not dissolve; but a calx of lead, probably produced by the violent action of the acid, and which, by the addition of phlogiston, may be exhibited in its metallic form. The quantity of this calx depends much upon the action of the acid upon the ore; if that action is violent, the calx is in greater abundance

than if it be moderate; and I am not certain whether the experiment might not be so managed, that there would be little or no calx remaining; that is, a given quantity of ore might be so dissolved in the acid of nitre, that nothing would remain undissolved except the sulphur. But I have not yet perfectly satisfied myself as to the constituent parts of lead ore. I am certain that it contains lead and sulphur, a liquid and air: of the existence of the three first there can be no doubt, from what has been said, and the air is rendered beautifully apparent by the following experiment.

Let some lead ore be reduced into a fine powder, put it into a narrow-bottomed ale glass, fill the glass three parts with water, drop into the water a portion of the strong acid of nitre; you may judge of the requisite quantity by seeing the solution commence, and you will observe the ore universally covered with bubbles of air, these will buoy the ore up in large tufts to the surface, and the air will continue to be separated from the ore till the acid becomes saturated with the lead. The salt arising from the union of the nitrous acid to the lead often appears crystallized upon the surface of the menstruum in this experiment; and if, when the menstruum is in that state, a little fresh acid be added, the salt instantly crystallizes and falls down to the bottom of the glass,

glass, the acid having absorbed the water which held it in solution. When lead is dissolved in the manner here mentioned, by a very diluted acid of nitre, there is no appearance of sulphur upon the surface of the menstruum, there is found at its bottom a black matter which is the sulphur.

But though lead and sulphur, a liquid and air, are unquestionably constituent parts of lead ore, I do not take upon me to say, that they are the only constituent parts: it is well known, that, during the smelting of lead ore, a third part or more of its weight is somehow or other lost, since from one and twenty hundred weight of ore they seldom obtain above fourteen hundred weight of lead. What is lost partly consists of a scoria which floats upon the surface of the lead during the operation of smelting, and partly of what is sublimed up the chimney and diffipated in the air. The scoria, I apprehend, would be very little even from a ton of ore, if the ore was quite free from spar: it is the spar which is mixed with the ore that constitutes the main portion of the scoria. I have in my possession a solid mass of scoria, which accidentally flowed out from a smelting furnace, and which in colour and consistency perfectly resembles grey lime-stone; it receives a polish as fine as marble, and it might, perhaps, with advantage be cast into molds for paving stones,

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chimney



chimney pieces, and other matters. It arises from the spar mixed with the ore, and, by the addition of fusible spar to the ore during its fusion, its quantity might be increased at no great expence, in any proportion. That part of the ore which is sublimed and dispersed in the air, consists partly of the sulphur which is consumed, and partly of lead; this sublimed lead attaches itself in part to the sides of the chimney of the smelting furnace; the rest of it flies up into the air, from whence it falls upon the ground, poisoning the water and herbage upon which it settles. This sublimed lead might be collected either by making it meet with water, or with the vapour of water during its ascent, or by making it pass through an horizontal chimney of a sufficient length.

It is not easy to determine with precision the quantity of this sublimed lead; a general guess, however, may throw some light upon the subject. They usually at a smelting house work off three tons, or sixty hundred weight, of lead ore every twenty-four hours; the sulphur contained in sixty hundred weight of ore we will suppose to be seven hundred weight, and the lead to be forty hundred weight; the air, liquid, scoria, and sublimed lead, must together, upon this supposition, amount to thirteen hundred weight; now, admitting three hundred weight of the thirteen to be sublimed lead, it is  
evident

evident that, could it be collected, there would be an annual saving at each smelting house of above fifty tons, which, supposing it to be worth four pounds *per* ton, would amount to above two hundred pounds a year. The price and quantity of lead sublimate here assumed are probably both of them below the truth; but my end is answered in giving this hint to persons engaged in the smelting business.

The following experiments, though upon a different subject, may not be unacceptable to the lovers of chemistry, as I do not remember to have any where met with them. I trouble the Society with a relation of them at this time, that I may not hereafter intrude upon their leisure.

It is commonly known, that the surface of melted lead becomes covered with a pellicle of various colours. I undertook some experiments in the course of last winter, with a view to ascertain the order in which the colours succeeded each other. The lead which lines the boxes in which tea is imported from China happening to be at hand, some of it was melted in an iron ladle; but I was much surprized to find that its surface, though it was presently covered with a dusky pellicle, did not exhibit any colours. Imagining that the heat was not sufficiently strong to render the colours visible, the fire was urged till the ladle became red-hot, the calcined pellicle  
upon.

upon the surface of the lead was red-hot also, but it was still without colour. The same parcel of lead was boiled in a crucible for a considerable time; during the boiling a copious steam was discharged, and the surface of the lead, as is usual, became covered with a half vitrified scoria. The lead which remained unvitrified was then examined, and it had acquired the property of forming a succession of coloured pellicles during the whole time of continuing in a state of fusion.

Another portion of the same kind of lead was exposed to a strong calcining heat for a long time; the part which remained uncalcined did, at length, acquire the property of exhibiting colours sufficiently vivid.

These experiments induced me to conclude, that the Chinese lead was mixed with some substance from which it was necessary to free it, either by sublimation or calcination, before it would exhibit its colours. It would be useless to mention all the experiments which I made before I discovered the heterogeneous substance with which I supposed the Chinese lead was mixed. At last I hit upon one which seems fully sufficient to explain the phenomenon. Into a ladle full of melted Derbyshire lead, which manifested a succession of the most vivid colours, I put a small portion of tin, and observed, that as soon as the tin was melted, and mixed with the lead, no  
more

more colours were to be seen. I do not know precisely the smallest possible quantity of tin, which will be sufficient to deprive a given quantity of lead of its property of forming coloured pellicles, but I have reason to believe, that it does not exceed one five thousandth part of weight of the lead.

Derbyshire lead, which has lost its property of exhibiting colours by being mixed with tin, acquires it again, as is mentioned of the Chinese lead, by being exposed to a calcining heat for a sufficient time; the tin it is supposed being separated from the lead by calcination before all the lead is reduced to a calx.

Some calcined Chinese lead was reduced to its metallic form by burning some tallow over it. The reduced lead gave, when melted, coloured pellicles; the calx of tin, which we suppose to have been mixed with the calcined lead, not being so easily reducible as that of lead.

I find that zinc is another metallic substance which has the same property as tin with respect to the depriving lead of its power of forming coloured pellicles; but it does not, I think, possess this power in so eminent a degree as tin. I put small portions of bismuth also into melted lead, but the lead still retained its quality of forming colours. I melted together some silver and lead, but the lead did not thereby lose its power of forming colours.

A little tin added to a mixture of lead and bismuth, or to a mixture of silver and lead, immediately takes away from the respective mixtures the faculty of forming coloured pellicles.

This quality of tin has hitherto, as far as I know, been unobserved; but every new fact, relative to the actions of bodies one upon another, ought to be recorded. The change produced in lead by the admixture of a small portion of tin is much felt by the plumbers, as it makes the metal so hard and harsh, that it is not without difficulty they can cast it into sheet lead. If their old lead does not work so willingly, nor exhibit colours so readily, as new lead, they may refer the difference to the small quantity of tin contained in the folder, from which old lead can seldom be thoroughly freed.

With respect to the order in which the colours succeed one another upon the surface of melted lead, it seems to be the following one; yellow, purple, blue, yellow, purple, green, pink, green, pink, 'green. Upon exhibiting the bright surface of melted lead to the air, I have often observed these ten changes to follow one another in a more or less rapid succession, according to the degree of heat prevailing in the lead. If the heat is but small, the succession stops before it has gone through all the changes; but with the greatest heat I did not observe any  
further

further variation. All the colours are very vivid, and each seems to go through all the shades belonging to it before it is changed into the next in order.

The formation of these colours may be explained from what has been advanced by Sir ISAAC NEWTON, and illustrated by the very ingenious experiments of Mr. DELAVAL, relative to the size of the particles constituting coloured bodies.

